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Interior air of road vehicles —

Part 12:
Artificial leather made from PVC or Polyurethane— Specification and methods for the determination of fogging characteristics of trim materials in the interior of automobiles

ICS: 13.040.20; 43.020

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Introduction

Artificial leather is widely used in car interiors. Test methods to determine the fogging characteristics of PVC or polyurethane artificial are described in this document.

The test methods are also be applicable to determine other materials and finished products which may be used for trim materials.

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Interior air of road vehicles —

Part 12:
Artificial leather made from PVC or Polyurethane— Specification and methods for the determination of fogging characteristics of trim materials in the interior of automobiles

1 Scope

This document specifies test methods which are intended to determine the fogging characteristics of PVC or polyurethane artificial leathers that are used as trim materials in the interior of motor vehicles.

The methods may also be applicable to fluid, paste, powdered or solid raw materials which are the basis for such trim materials or from which the materials are manufactured. The methods may also be applicable to other materials and finished products.

The procedures are applicable to the measurement of fog condensate on glass surfaces within the limits of the test conditions. These tests will not measure or cannot measure accurately those cases in which:

— the surface tension of the condensate is low, resulting in early coalescing into a thin transparent film;

— the condensate is present in such a large quantity that the droplets coalesce and form a heavy oily/clear film (this heavy film gives false readings).

In such cases, the gravimetric method is preferred.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2813, *Paints and varnishes — Determination of gloss value at 20°, 60° and 85°*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

A test specimen is heated in a glass beaker. Any volatile constituents are condensed on either a cooled glass plate or a disc of cooled aluminium foil.

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The fogging value *F* is calculated as the quotient, in percent, of the gloss value for the glass plate with fogging condensate and the gloss value of the same glass plate without fogging condensate. The gloss values are measured in accordance with ISO 2813.

The mass of the condensable constituents *G* is given by the difference between the masses of the aluminium foil disc before and after fogging.

5 Apparatus and materials

5.1 Thermostatically controlled bath or chamber, typically designed to operate at up to 130°C. Safety devices shall be fitted to prevent overheating. The circulation system, the bath or chamber capacity and the heating system shall be such that the temperature can be kept constant to within ± 1,0 °C throughout the bath or chamber.

NOTE To keep the temperature constant it is recommended that the agitation of the bath or chamber should de carried out at a slow uniform speed.

The bath or chamber shall be designed so that, after placing the beakers (5.5) in the bath or chamber, the temperature does not drop more than 5 °C, and the test temperature is regained after no more than 20 min. The minimum distance between the beakers and the walls shall be 30 mm. The oil bath shall be equipped with a device indicating the distance between the fluid and the lower surface of the glass plate (5.3). This distance shall be (60 ± 2) mm. When using an air chamber, the beaker shall be placed so that the entire beaker up to the rim is placed inside the chamber to be heated.

See Annex A for comparison of data obtained from an oil bath and air chamber with correct positioning of the beakers to give similar results.

5.2 Thermal-transfer fluid, liquid or air, for the thermostatically controlled bath or chamber (5.1). The fluid shall be temperature-stable and preferably water-soluble for easier cleaning. A suitable fluid is a modified polyhydric aliphatic alcohol.

5.3 Float-glass plates, of residental or windshield window quality, for condensation of the fogging, thickness (3 ± 0,3) mm, either square with minimum dimensions of (110 × 110) mm or circular with a diameter of 103 mm. The gloss values of all the plates used shall be the same to within ± 2 % units. The tin and non-tin surfaces of the plate shall be identified, and the identification mark shall be placed on the tin surface. The non-tin surface is the test surface that faces the test specimen during the test.

The tin and non-tin surface of the glass plate can be identified by viewing the surface in a darkened room under a UV light at 254 nm wavelength. The tin surface will fluoresce when it is exposed to the UV light.

5.4 Cooling plates, designed to be placed on the glass plate (5.3) to keep them cool. The cooling plate shall be hollow and made of corrosion-resistant metal, with the side facing the glass plate made of aluminium. They shall have two cooling-water connections located so that the cooling water flows through the whole of the interior of the plate. The surface in contact with the glass plate shall be flat. When using liquid for thermal transfer fluid, the mass of a cooling plate filled with water shall be at least 1 kg, to overcome the buoyancy of the beaker (5.5) in the bath. The whole of the weight of the cooling plate shall rest on the beaker. A separate cooling plate shall be used for each beaker.

The cooling plate and the associated water thermostat shall be designed so that the mean water temperature is 21 °C and the difference in temperature between the inlet and outlet does not exceed 1 °C.

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5.5 Flat-bottomed beakers, of heat-resistant glass, minimum mass 400 g, with the dimensions shown in Figure 1.

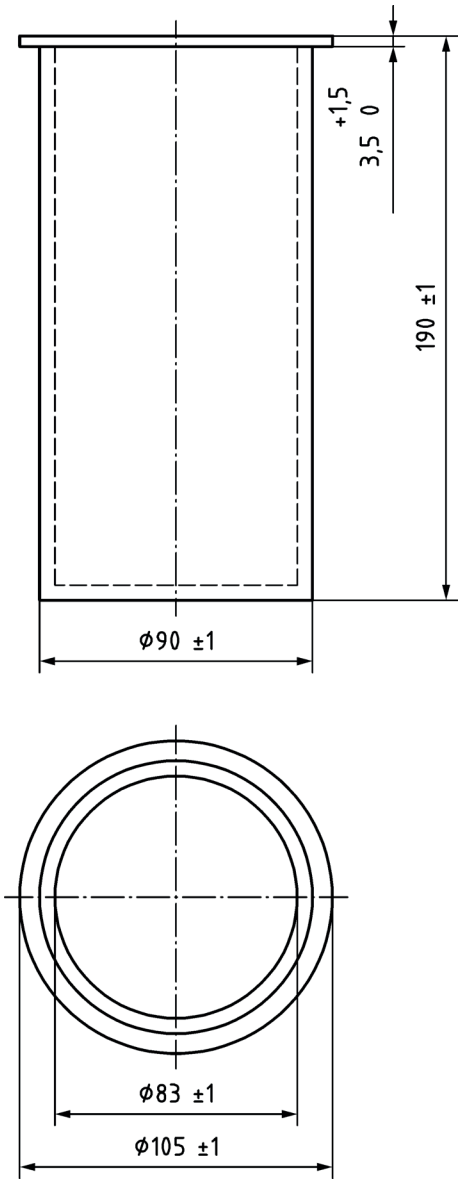

Figure 1 — Glass beaker; Dimensions in mm

5.6 Reference liquid, diisodecyl phthalate (DIDP).

Example of a vendor for DIDP reference liquid purchase is noted in the footnote[1)]. An analytical reagent grade or equivalent can also be used. Results of DIDP may differ by vendor and shall be verified.

See Annex B for examples of DIDP results of different vendors.

5.7 Metal rings, external diameters 80 mm, internal diameter 74 mm, height 10 mm and mass (55 ± 1) g, made of corrosion-protected steel, to keep the test piece flat.

1) DIDP reference liquid can be obtained from: SP Technical Research Institute of Sweden, Chemistry and Materials Technology, Box 857, SE-501 15 Borås, Sweden, Fax: +46 33 10 33 88, E-mail: info@sp.se. This information is given for the convenience of users of this standard and does not constitute an endorsement by ISO of their products.

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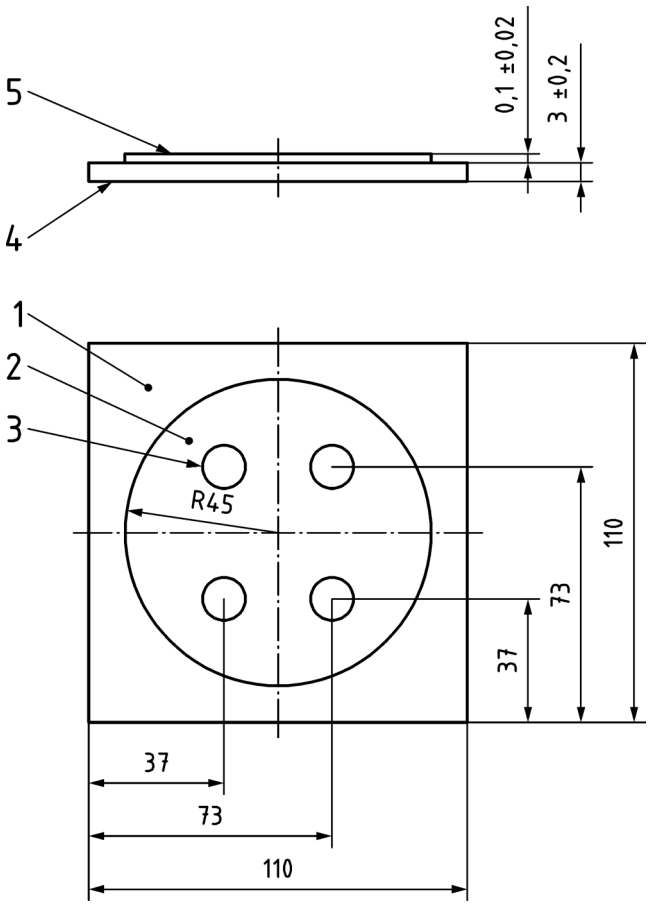
5.8 Sealing rings, of silicone- or fluoro-rubber, L-shaped or circular in cross-section, inner diameter 90 mm to 95 mm, thickness 2 mm to 4 mm and hardness 50 IRHD to 70 IRHD.

5.9 Filter paper, with a diameter of 110 mm and a mass per unit surface area of 90 g/m^2.

5.10 Aluminium foil discs, thickness (0,02 ± 0,01) mm, circular with a diameter of (103 ± 1) mm, or square with minimum dimensions of (110 x 110) mm. The shape of the aluminium foil shall not matter if it has been verified that there is no effect on results. Store the prepared aluminium foil disc in a desiccator (5.18) to avoid condensation and contamination.

5.11 Gloss meter, with a 60° incident beam and 60° measurement beam in accordance with ISO 2813.

5.12 Spacer, designed to prevent contact with the condensate on the glass plate during gloss meter measurements, made of a suitable material such as paper or plastic with circular hole for the measurements. The thickness of the spacer shall be (0,1 ± 0,02) mm (see Figure 2).



Key

1 float glass plate
2 spacer
3 circular holes for the measurements
4 tin surface of glass plate
5 non-tin surface of glass plate

Figure 2 — Example of a spacer on top of glass plate; Dimensions in mm

5.13 Dishwasher, preferably connected to a deionized-water supply and capable of being operated at 80 °C.

5.14 Balance, with scale divisions of 0,01 mg.#

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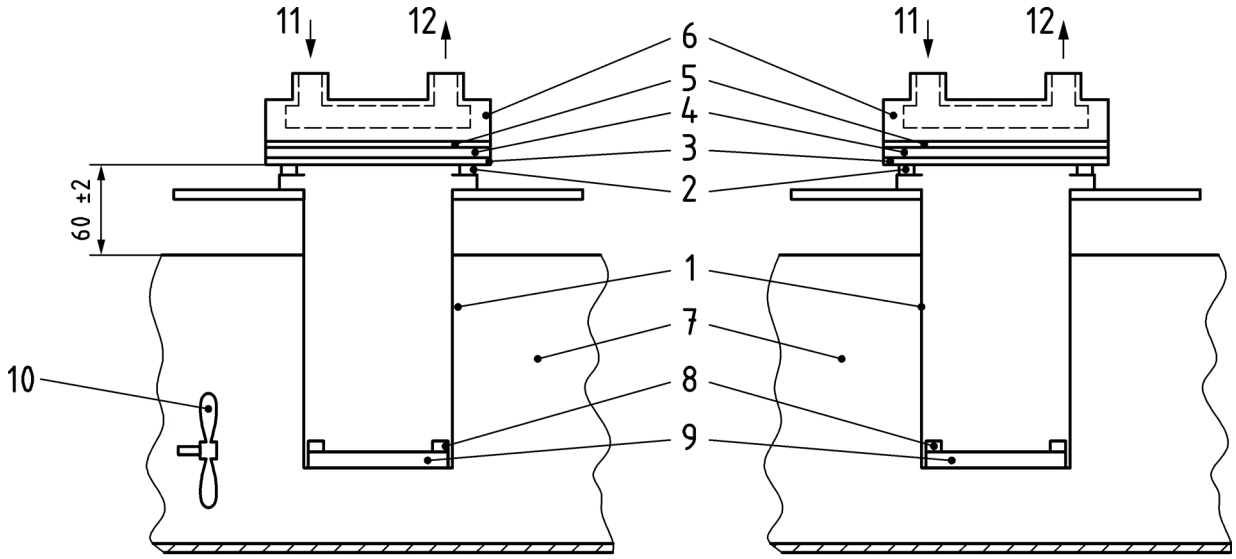
5.15 Glass-cleaning detergent of a neutral or acidic type.

5.16 Polyethylene gloves.

5.17 Tongs.

5.18 Desiccator, with suitable drying material.



Key

A	Oil bath	B	Air chamber
1	beaker	7	thermal transfer fluid (liquid or air)
2	sealing ring	8	metal ring (if necessary)
3	aluminium foil disc (determination of mass of condensable constituents G test only)	9	test piece
4	float glass plate	10	agitating fan
5	filter paper	11	cooling-water inlet
6	cooling plate	12	cooling-water outlet

Figure 3 — Example of a test apparatus

6 Sample preparation

In the case of finished products, cut circular test specimen with a diameter of (80 ± 1) mm from the sample. The thickness of the test specimens can be up to 10 mm. Reduce thicker materials by removing material from the underside until a 10 mm thickness is reached (the underside is the side facing away from the side which is visible in the vehicle). If other test specimen dimensions are required, these may be as given in the product specification or as agreed between the interested parties.

NOTE 1 Sample indicates the material before cutting. Test specimen indicates the sample cut into the required size for sampling.

Take two test specimens for the determination of the fogging value *F* and another two for the determination of the mass of the condensable constituents *G*.

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7 Conditioning

Unless otherwise specified than in Table 1, condition all test specimens at 23 °C and 50 % RH for at least 1 day.

Table 1 — Example conditioning times

Material	Conditioning time day
PVC artificial leather	1
Polyurethane artificial leather	1

8 Procedure

8.1 Cleaning

8.1.1 General

Only touch the beakers (5.5) on the outer surfaces. Do not touch the glass plates (5.3) or the metal rings (5.7) with bare hands; use gloves (5.16) or tongs (5.17).

After the glass plates have been cleaned and dried, make a visual check that the plates are free of scratches and other defects; if they are not, discard them.

After cleaning, store all items, with the beakers upside-down, in a dust-free environment at room temperature until the measurements are made.

8.1.2 Cleaning with a dishwasher

Wash the sealing rings (5.8), beakers and metal rings twice in a dishwasher (5.13) with glass-cleaning detergent (5.15). If the dishwasher is not connected to a deionized-water supply, rinse the cleaned equipment in deionized water.

Prior to each use, clean all glass plates twice in the dishwasher at a temperature of 80 °C using a glass-cleaning detergent. If the dishwasher is not connected to a deionized-water supply, rinse the cleaned glass plates in deionized water at room temperature and dry them in an upright position.

It is recommended that the glass plates are not reused more than a few times, since microscopic scratches may affect the rate of deposition of any vapours and hence the reproducibility of the method. Discard any glass plates that have surface scratches or abraded spots.

Tests have shown that it is very important to use a neutral or acidic detergent as alkaline detergent affects the glass surface and the fogging value calculated from gloss values increases.

8.1.3 Manual cleaning

When a dishwasher is not available, cleaning can be done with a hand-washing procedure using a neutral or acidic detergent.

8.1.4 Polishing of glass surface

Tests have shown that polishing the surface of the glass plate to make it hydrophilic is important to obtain stable fogging test results (see Annex C).

8.2 Reference tests

Carry out a reference test to determine the fogging value F of the reference liquid DIDP (5.3) regularly as required, or at least every 6 months. For this purpose, add (10 ± 0,1) g of DIDP to a beaker, taking care not to

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moisten the inner wall of the beaker. Place the beaker with the DIDP in the bath or chamber (5.1). After the test period of (180 ± 3) min at the oil bath or air chamber temperature of (100 ± 1,0) °C, the fogging value shall be within 50 to 98, and the variation coefficient shall be within ±5%. If this is not the case, check the test conditions.

Carry out the same procedure with DIDP when determining the mass of the condensable constituents *G*. After the test period of (16 ± 0,2) h at the oil bath or air chamber temperature of (100 ± 1,0) °C, the mass of the condensable constituents shall be within ±0,25 mg as compared with the control value. If this is not the case, check the test conditions.

It is very important not to moisten the walls of the beaker with the DIDP when handling the beaker, as this will give a larger area with DIDP and a higher test result.

8.3 Placement of test specimens

Insert the test specimen into beakers (one test specimen per beaker). Do not touch the test specimens with bare hands.

Place the test specimens so that the visible side, i.e. the side facing the vehicle interior, faces upwards. If necessary, to prevent rolling and warping of the test specimens, place a metal ring on each.

8.4 Measurements prior to the fogging test

When gloss values are to be determined, calibrate the gloss meter (5.11) in accordance with the manufacturer's operating instructions. For this purpose, place a glass plate, with the non-tin side up, on a matt-black filter paper backing and place a spacer on the glass plate. Make markings on the spacer for the edges of the gloss meter. Place the gloss meter against these markings. The measurement spots shall be at a distance of (25 ± 5) mm from the centre of the glass plate. Take four readings R_{01} to R_{04} with the-gloss meter aligned parallel to the plate edges (or the tangents to the plate edges in the case of circular plates), rotating the gloss meter by 90° between each reading.

Since the measured gloss meter values before and after the fogging test are to be linked in pairs, the measurement geometry must be the same in each case. The glass plates must therefore be marked so that they can be positioned in exactly the same way before and after the fogging test.

When the mass of the condensable constituents is to be determined, use aluminium foil discs. Use polyethylene gloves when handling the discs. Take care not to crease the discs. A recommended method of preparing the discs is to cut them out by stamping from several layers of aluminium foil alternating with layers of paper edge-welded to the foil. Take care to avoid contaminating the discs when cutting them out. Use the depression produced in the discs by the stamping procedure to give an improved seal with the sealing ring. Determine the mass G_0 of each disc to ± 0,02 mg.

8.5 Fogging test

8.5.1 Set-up

Place a sealing ring on top of each loaded beaker. For the determination of the fogging value *F*, place a clean glass plate, with its non-tin surface of known R_0 value facing downwards, on top, covering the beaker. For the determination of the mass of the condensable constituents *G*, place a weighed aluminium disc (5.10), bright side downwards, on the sealing ring, followed by a clean glass plate.

Place the beakers prepared in this manner in the thermostatic bath or chamber held at (100 ±1,0) °C.

NOTE 1 Other temperatures may be agreed upon by the interested parties.

Place a filter paper (5.9) on each glass plate (to prevent scratching of the surface of the glass plate), followed by a cooling plate (5.4).

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Set the temperature of the cooling water at (21 ± 1) °C.

NOTE 2 Other temperatures my be agreed upon by the interested parties.

NOTE 3 In case of using a liquid for thermal transfer fluid, when changing the test temperature, check the level as the volume of the liquid changes with temperature.

8.5.2 Determination of fogging value *F*

Keep the beakers in the thermostatic bath or chamber for a period of (180 ± 3) min.

NOTE 1 Other periods may be agreed upon by the interested parties.

Then raise the glass plates without touching the fogging condensate and store in a horizontal position, with the fogging condensate upwards, in a dust- and draught-free environment at (23 ± 2) °C and below 55 % RH. Do not expose the glass plates to direct sunlight.

Carry out the measurement of the gloss meter value after a storage period of (60 ± 6) min.

NOTE 2 Other periods may be agreed upon by the interested parties.

Prior to measuring the fogging condensate with the gloss meter, check visually that the condensate does consist of droplets, and does not consist of a continuous film or contain crystals or other structural features. Do not measure gloss meter values of such condensates since they give misleading results. If such condensates are formed, mention this fact in the test report. If necessary, repeat the test.

Calibrate the gloss meter. Then place the glass plate on the black filter paper backing, and the spacer on the glass plate.

Place the gloss meter over the guide markings and take four readings R_{11}, R_{12}, R_{13} and R_{14}.

Measure the gloss values for two test specimens. If the fogging values obtained deviate by more than 10 % from the mean value test a further two test specimens and calculate the mean value of only those which are within ± 10 % of the mean.

8.5.3 Determination of mass of condensable constituents *G*

Allow the beakers to remain in the thermostatic bath or chamber for (16 ± 0,1) h. After this period, carefully remove the aluminium discs, on their sealing rings, and store them with the fogged side up in a desiccator for 3,5 h to 4 h. Do not over-fill the desiccator. Do not expose the discs to direct sunlight in the desiccator. Determine the mass G_1 of each fogged disc to within 0,01 mg.

Carry out measurements on two test specimens. If the masses of the condensable constituents deviate by more than 10 % from the mean value, test a further two test specimens and calculate the mean of the results obtained for all four test specimens.

9 Expression of results

9.1 Fogging value

To calculate the fogging value *F*, first calculate the fogging value F_j for each glass plate using equation (1)

$$F_j = \left(\frac{R_{11}}{R_{01}} + \frac{R_{12}}{R_{02}} + \frac{R_{13}}{R_{03}} + \frac{R_{14}}{R_{04}} \right) \times \frac{100}{4} \quad (1)$$

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where

F_j is the fogging value for the jth plate, in %;

R_{11} to R_{14} are the gloss meter readings for the fogged plate, in GU;

R_{01} to R_{04} are the gloss meter readings for the unfogged plate, in GU.

The fogging value F, in %, is the mean of the F_j values. Report F to the nearest whole number.

9.2 Mass of condensable constituents

To calculate the mass of the condensable constituents G, first calculate the mass of condensable constituents G_j on each aluminium disc using equation (2)

$$G_j = G_1 - G_0 \quad (2)$$

where

G_j is the mass of the condensable constituents on the jth disc, in mg;

G_0 is the mass of the disc before the test, in mg;

G_1 is the mass of the disc after the test, in mg.

The mass of the condensable constituents G, in mg, is the mean value of the G_j values. Report G to the nearest 0,1 mg.

10 Test report

The test report shall include the following particulars:

a) a reference to this document including its year of publication i.e ISO xxxx; XXXX(year);

b) the parameter measured, i.e. the fogging value F or the mass of the condensable constituents G;

c) all details necessary for complete identification of the test samples;

d) the temperatures and times used for the test;

e) the test results, i.e.

fogging value:	individual values F_j
	arithmetic mean F
	number of test specimens
	control value (DIDP);
or	
condensable constituents:	individual values G_j
	arithmetic mean G
	number of test specimens
	control value (DIDP);

f) any special observations, e.g. details of condensates that could not be measured, large droplets, film formation, crystal formation or running condensates;

g) any deviations from the standard test procedure;

h) the date of the test.

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Annex A
(informative)

Comparison of air chamber and oil bath test results

A.1 General

A.1.1 Method-A was performed using DIDP with a specified reflectance criteria (77 ± 3) % obtained from VVC (Verson Vlies Courcier s.a), using both the the oil bath and air chamber.

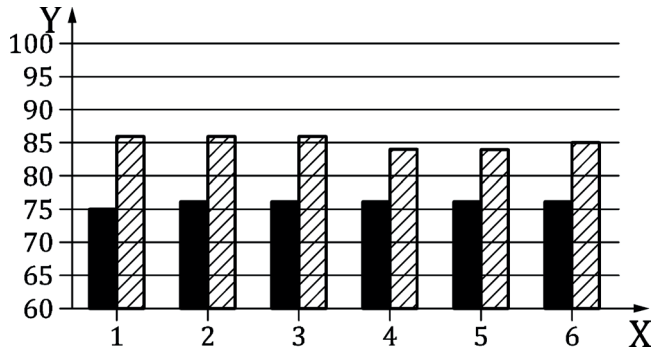
A.1.2 Test condition [1] was in accordance with DIN75201, set with a distance of 60 mm from the top of the thermostatically controlled fluid to the bottom surface of the glass plate.

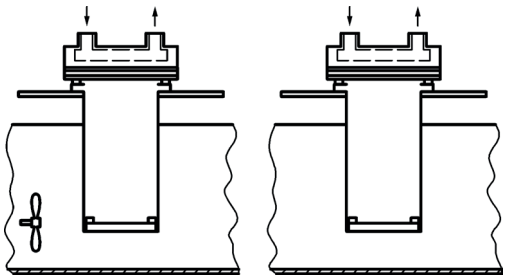
A.1.3 Test condition [2] was performed with the oil bath set with a distance of 60 mm from the top of the thermostatically controlled fluid to the bottom surface of the glass plate, while the air chamber was set with the rim of the beaker seated directly on top of the air chamber oven.

A.2 Results

Under test condition [1] , there was a large difference in reflectance values between the oil bath and the air chamber.

Under test condition [2], the results of oil bath and air chamber were equivalent. In the oil bath, the distance between the surface of the oil and the bottom of the glass plate is 60 mm, but the lid on the oil bath helps maintain the heat of the beaker. With the air chamber, the ambient room temperature affects the part of the beaker not residing in the air chamber, resulting in the decreased temperature inside the glass beaker.





Key

X Beaker #

Y F Value (%)

oil bath

air chamber

Figure A.1 — Test condition [1] 60 mm distance set between top of thermostatically controlled fluid/air to bottom surface of glass plate

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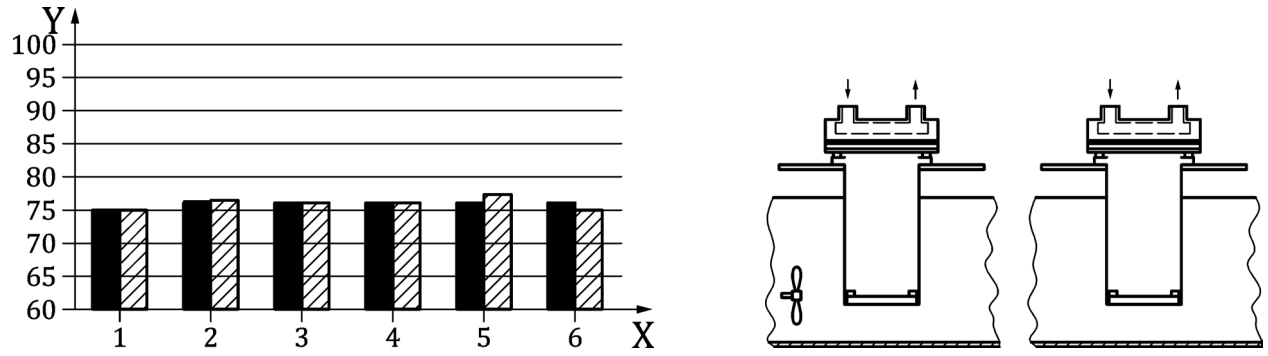

Key

X Beaker #

Y F Value (%)

oil bath

air chamber

Figure A.2 — Test condition [2] 60 mm distance set between top of thermostatically controlled fluid to bottom surface of glass plate with oil chamber only

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Annex B
(informative)

Confirmation of fogging test bath accuracy by DIDP

B.1 General

DIDP (SP Technical Research Institute of Sweden) specified in DIN 75201:2011 is difficult to purchase in some countries. The following tests were conducted for the pupose of finding an appropriate reference value using other available vendors of DIDP to calibrate the fogging test equipment.

Tests were conducted using DIDP from four different vendors. DIDP from vendor A and B are specifically produced for fogging test purposes while those from vendor C and D are general reagents. Tests were performed for both Method-A (reflectance) and Method-B (weight).

B.2 Results

The results of the actual test result values are given in Table B.1 and Table B.2.

Based on the DIDP test results of the four vendors, the criteria for Method-A and Method-B were established. Since the measured values of each DIDP varied, the coefficient of variation was set from the test results. From the actual DIDP values of the four vendors, the coefficient of variation of Method-A was within ± 5 %. The mass of condensed components of Method-B was within ± 0,25 mg of the control value.

Table B.1 — Actual result values of Method-A

DIDP	Vendor A	Vendor B	Vendor C[1)]	Vendor D
Mean of Reflectance Fj value	70,5	75,7	58,3	67,1
Standard Deviation sj	1,2	1,6	2,3	3,3
Coefficient of variation v (%)	1,7	2,2	4,0	4,9
1) Test of Vendor C was performed without filter paper placed between glass plate and cooling plate.				

Table B.2 — Actual result values of Method-B

DIDP	Vendor A	Vendor B	Vendor C[1)]	Vendor D
Mean of Mass G (mg) value	0,72	0,73	1,52	1,25
Standard Deviation sG	0,07	0,05	0,06	0,07
G (mg)	+0,06 -0,10	+0,04 -0,08	+0,07 -0,09	+0,05 -0,13
1) Test of Vendor C was performed without filter paper placed between glass plate and cooling plate.				

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Annex C
(informative)

Effect of polishing glass plate surface

C.1 General

C.1.1 It is desirable to clean the glass plate to make the surface hydrophilic. Water repellency can cause water droplets (condensation water) to form on the surface of the glass plate. The components adhering to the glass surface can be washed away by the water droplets, causing unstable fogging values.

C.1.2 After cleaning, glass plates are polished to make the surface hydrophilic.

C.2 How to polish the glass plate (example)

1) Prepare a dishwasher-cleaned or manually cleaned glass plate.

2) Prepare abrasive powder (e.g., calcium carbonate or cerium oxide) and a soft cloth.

3) Apply calcium carbonate or cerium oxide dispersed in water onto the glass plate.

4) Polish the glass plate surface with a cloth. The use of an electric polisher can be effective.

NOTE Typical particle size of a polishing powder is under 200 µm. Note that larger sizes could cause scratching.

Key
1 before polishing
2 after polishing

Figure C.1 — Effect of glass plate polishing

C.3 Results

C.3.1 Fogging values were compared using a glass plate cleaned manually with a neutral detergent and a glass plate polished with cerium oxide.

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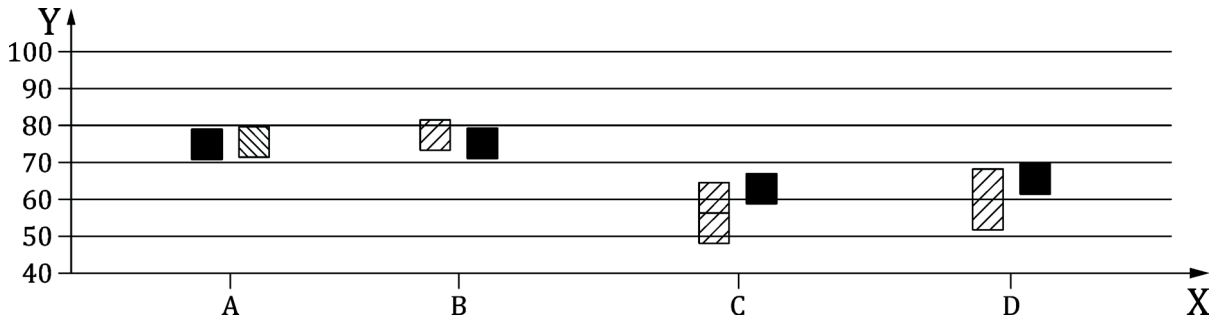
C.3.2 Results showed that glass plate polished with cerium oxide had more stable results of fogging values, standard deviation, and the coefficient of variation compared to plates without polishing. Washing with a dishwasher also showed effective results.



Key

X DIDP vendor

Y Fj(%)

not polished

polished

dishwasher

Figure C.2 — Effect of glass polishing on F_j (%) results

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Bibliography

[1] ISO 6452, *Rubber- or plastics-coated fabrics — Determination of fogging characteristics of trim materials in the interior of automobiles*

[2] ISO 17071, *Leather — Physical and mechanical tests — Determination of fogging characteristics*

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